

Experimental report

26/01/2016

Proposal: 5-31-2443

Council: 4/2015

Title: Search for magnetic order in epsilon-iron at 20 GPa

Research area: Physics

This proposal is a continuation of 5-31-1676

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Samples: Fe

Instrument	Requested days	Allocated days	From	To
D20	3	3	30/11/2015	03/12/2015

Abstract:

In this proposal we ask for beamtime to collect high pressure neutron diffraction data of epsilon-iron at low temperatures. It is a continuation of experiment 5-31-1676 (June 2007) with a significantly improved experimental setup. Epsilon-iron is the high pressure phase of iron which is stable beyond 16 GPa (160 kbar). It has been speculated since decades that this phase might be magnetic. This would have important implications for the Earth Sciences (the Earth's core is made of epsilon-iron), but also for fundamental solid state physics since epsilon-iron is superconducting below $T_c=2$ K.

Experimental report "Search for magnetic order in epsilon iron at 20 GPa" (proposal nr. 73344, experiment nr. 5-31-2443)

Main proposer: S. Klotz

The purpose of the experiment was to search for magnetic ordering in ϵ -Fe, the high-pressure phase of iron stable beyond ~ 16 GPa [1]. *Ab-initio* calculations predict an anti-ferromagnetic ground state [2], and there is various experimental evidence that ϵ -Fe is paramagnetic at 300 K [3]. The question has considerable importance for both fundamental physics as well as for Geosciences. Epsilon-iron (hcp) is a superconductor [4], and this phase is the main constituent of the Earth's core.

This request was already the subject of our previous beamtime 5-31-1676 in May 2007. Although these measurements were rather successful, the patterns of ϵ -Fe were contaminated by signal from the anvils which hampered the analysis of the data. Also, the lowest temperature achieved was 3.6 K. Recent technical developments showed that much lower temperatures of 1.8 K could be achieved with only minor modifications of the same equipment. A revisit of such measurements with improved experimental conditions was hence highly desirable.

The experiment was carried out using the ILL Paris-Edinburgh pressure cell with its dedicated cryogenic equipment [5], sintered diamond anvils and encapsulating TiZr gaskets allowing a sample volume of approximately 8 mm^3 . The design and shielding were improved compared to 5-31-1676 to reduce considerably contaminant scattering from the anvils. The initial room temperature compression to 100 tonne gave patterns of pure ϵ -Fe (plus diamond) with refined unit cell parameters of $a=2.44232(20) \text{ \AA}$ and $c=3.92918(39) \text{ \AA}$, i.e. $V=20.297(3) \text{ \AA}^3$, hence a pressure of 21.3 GPa according to the Vinet-Rydberg equation-of-state of Ref. [6] ($V_0=11.214 \text{ \AA}^3/\text{atom}$, $B_0=163.4 \text{ GPa}$, $B'_0=5.38$). After cooling at constant load, the refinements of the patterns at 1.79 K (Fig. 1) give $a=2.43873(22) \text{ \AA}$ and $c=3.91908(40) \text{ \AA}$, i.e. $V=20.186(3) \text{ \AA}^3$. This indicates a pressure of 22.6 GPa using unpublished x-ray synchrotron data of ϵ -Fe obtained at 15 K [7] and the same type of equation-of-state with $V_0=11.207 \text{ \AA}^3/\text{atom}$, $B_0=163.6 \text{ GPa}$ and $B'_0=5.33$ [7]. After decompression at ~ 200 K the anvils were recovered undamaged. A complete analysis of these results including the search for the potential presence of magnetism in ϵ -Fe will be given separately.

The measurements were hence highly successful and opened new experimental possibilities. The technical aspects of these experiments were published 6 weeks after the measurements [8].

References

- [1] D. A. Young, *Phase Diagrams of the Elements*, Univ. of California Press, 1991.
- [2] G. Steinle-Neumann, L. Stixrude, R. Cohen, PNAS 101, 33 (2004).
- [3] D. Jaccard, A.T. Holmes, Physica B **359**, 333 (2005); S. Gilder & J. Glenn, Science **279**, 72 (1998)
- [4] Shimizu et al., Nature **412**, 316 (2001).
- [5] E. Bourgeat-Lami et al., Physica B 385-386, 1303 (2006).
- [6] A. Dewaele, P. Loubeyre, F. Occelli, M. Mezouar, P. Dorogokupets, M. Torrent, Phys. Rev. Lett. 97: 215504-4 (2006).
- [7] A. Dewaele, ESRF Exp. Report HC-1679 (Jan. 2014), ESRF, Grenoble, France.
- [8] S. Klotz, Th. Strässle, B. Lebert, M. d'Astuto, Th. Hansen, High Press. Research 36 (2016)

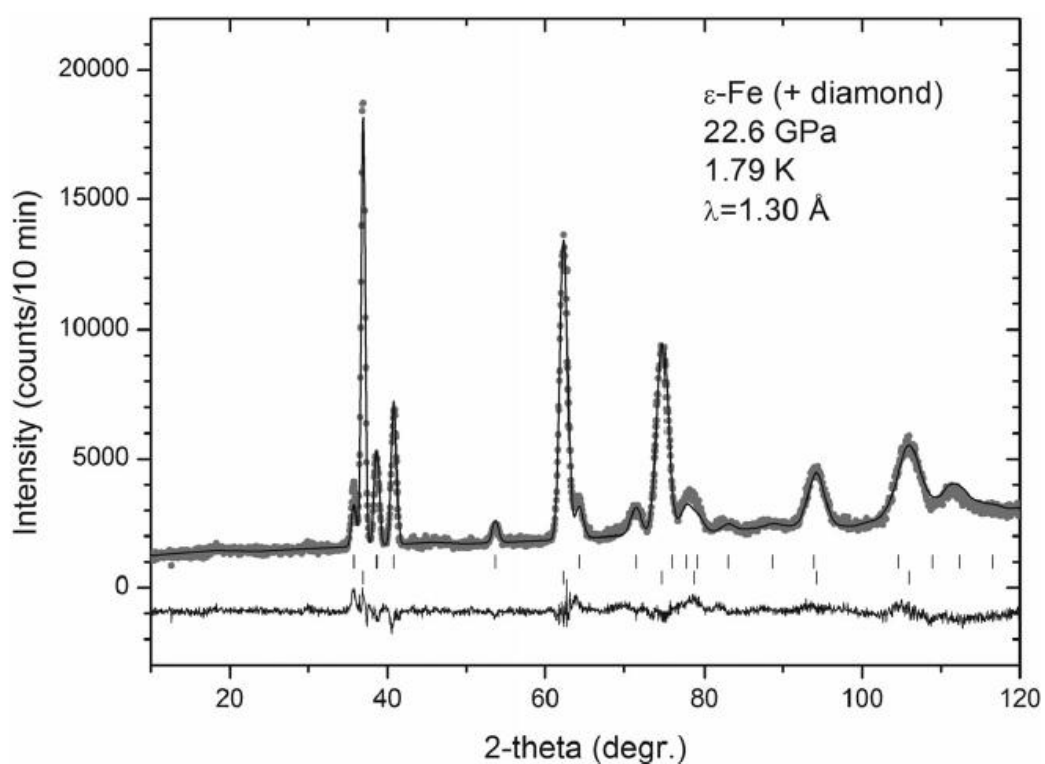


Figure 1: Diffraction pattern of ϵ -iron at 22.6 GPa and 1.79 K [8]. The line is a Rietveld fit to the data (dots), the difference curve is given below. Upper tick marks indicate ϵ -Fe peak positions, lower tick marks those of diamond from the anvils. The pattern represents raw data, i.e. no background was subtracted. The accumulation time is 10 min.

